CHAPTER 5

Holding Times and Preservation

5-1. Introduction.

The primary objective of this review is to ascertain the representativeness of the analytical data in the context of **preservation** and **holding time limits**[1]. **Holding times** for environmental samples are calculated from the dates of sample collection to preparation and analysis. (Refer to the glossary).

5-2. Acceptance Criteria.

a. The data package must clearly indicate the dates of all sample handling processes and the method of sample preservation. Holding time and preservation acceptance criteria and corrective actions are determined by method requirements and project DQOs. If holding time or preservation requirements are not specified for the project, use the published holding times and preservation requirements that are summarized in Table B-1 (Appendix B).

> Note: The preservation requirements and holding time limits were primarily taken from SW-846 and water methods (40 CFR, Part 136.3). Other sample preservation and holding time criteria may be more applicable (e.g., depending on the methods selected and matrices being tested).

b. Published holding time limits are generally considered maximum times that samples may be held before analysis and still be considered compliant with method guidelines, and typically apply to preserved samples.

> Note: Published holding times listed in environmental methods and regulations are not necessarily scientifically valid. However, use of alternative holding times may have a profound impact on the legal defensibility of results. It is recommended that published holding times be extended or shortened, if the client and regulators have agreed to other holding times for a particular project (e.g., based upon the chemistry of the method and holding time studies).

5-3. Evaluation.

a. Holding times and preservation are evaluated using the COC (Chain Of Custody) form, the laboratory's Cooler Receipt form (e.g., refer to the format of the USACE Cooler Receipt form), Case Narrative, sample preparation logs, and instrument run logs.

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[1]Sampling design (e.g., sampling locations) probably affects representativeness far more than any other factor. In the context of a full data usability assessment, the review described here should be viewed as a screening process to determine if the samples are *potentially* representative of the environmental matrices being sampled.

(1) Check sample holding times using the date of sample collection listed on the COC form with the dates of sample preparation and analysis reported on the laboratory's sample summary forms.

(2) Verify that digestion, extraction, or cleanup dates reported on the sample summary forms are identical to the dates listed on the sample preparation log sheets.

(3) Verify that the analysis dates reported on the sample summary forms are identical to those listed on the instrument run logs.

(4) Review the Case Narrative and Cooler Receipt form included in the data package to determine if all the samples were properly preserved and holding times were met. Note any problems that may have impacted the integrity of the samples (e.g., samples not maintained at 2^oC - 6^oC, aqueous VOC samples with head space, custody seals that are broken, and holding times that are not met). Verify that the pH of chemically preserved samples was checked and appropriate pH values were obtained.

b. If samples are properly preserved, holding time limits are met, and no problems with the samples are indicated in the Case Narrative, the laboratory's Cooler Receipt form, and the COC form, then assume that the physical integrity of the samples is acceptable.

c. If there are holding time violations or preservation problems, then the integrity of the samples may have been compromised. In the absence of information to the contrary, assume that holding time and preservation problems give rise to a *low* bias.

> Note: This assumption typically constitutes a conservative approach but will not always be appropriate. For example, the assumption will not be valid when degradation products are also target analytes. In particular, holding time noncompliances would presumably give rise to a low bias for pesticides such as DDT and Endrin, but to a high bias for associated degradation products such as DDE and Endrin aldehyde. In addition, when samples are held for an extended period of time, target analyte may leach or permeate into the storage containers, giving rise to a high bias. To address this potential problem, the representativeness of the method blanks would need to be evaluated. For example, if the method blank and samples were stored and analyzed together after the holding time limit, the absence of blank contamination in the method blank would suggest that leaching and permeation did not give rise to a high bias.

d. Distinguish *gross* holding time and preservation noncompliances from *marginal* noncompliances. Using the guidance presented in the "USEPA Contract Laboratory Program National Functional Guidelines For Organic Data Review," any holding time that is greater than twice the holding time limit is considered to be a *gross* holding time noncompliance. However, if holding time studies were performed, a gross holding time noncompliance would be defined on the basis of these studies.

e. Professional judgment is critical for evaluating holding time noncompliances. The evaluation of preservation problems is highly dependent upon the nature of the target analyte, the matrix, the method of analysis, and the sample handling procedures. For example, if aqueous samples for alkalinity were chemically preserved using pH adjustment, the analyses would be considered unusable. However, BNA soil sample results would not be rejected if a cooler temperature of 7°C were reported. Because of the time required to reach thermal equilibrium (relative to that required to ship the samples), samples may not cool during shipment to the 2–6°C acceptance range even when adequate refrigerant is placed in the cooler.

5-4.   Qualification.

The data qualification strategies presented in this section of the document are conservative in nature. A holding time noncompliance is assumed to give rise to a low bias.

5-4.1. Low Stability Target Analytes.

If there is a holding time noncompliance or a preservation problem for analytes that are known to readily volatilize or degrade in the matrix being tested (e.g., aqueous aromatic VOCs and hexavalent chromium), then qualify the results as follows:

   a. Qualify all nondetections with the R flag. For example, if large bubbles are reported in vials of the aqueous VOC samples or unpreserved aromatic VOC samples were analyzed beyond the limit, then qualify all nondetections with the R flag.

   b. At a minimum, qualify all detections with the J- flag. If an action level is specified and the analyte is detected at a concentration less than the action level, then qualify the detection with the X flag. Although the detection may be *qualitatively* reliable, it is not *quantitatively* reliable. Although the analyte was detected (despite the noncompliant holding time or preservation problem), the reported result potentially possesses a low bias and does not demonstrate that the analyte is actually present in the environmental sample at a concentration less than the action level.

5-4.2. High Stability Target Analytes

If there is a holding time or preservation noncompliance for analytes that are relatively stable in the matrix being tested (e.g., dioxins and trace metals such as lead in soils), then qualify the data as follows:

   a. If the holding time limit is *marginally* exceeded or the preservation problem does not appear to be significant, then qualify nondetections with the UN flag and detections with the J-flag.

   b. If there is a *gross* holding time or significant preservation problem, then qualify the data as discussed in Paragraph 5-4.1; namely, qualify nondetections with the R flag and detections with the J- flag or X flag.

Table 5-1
Data Qualification for Holding Time Noncompliance

Stability	Holding Time (t) [1]	Flag	Remarks [2]
Low	$t \leq$ HTL	None	Holding time limit is met.
	$t >$ HTL	R X J-	$y <$ MRL MRL $< y <$ AL $y >$ MRL and y $>$ AL
High	$t \leq$ HTL	None	Holding time limit is met.
	HTL $< t \leq 2$ HTL	UN J-	$y <$ MRL $y >$ MRL
	$t > 2$ HTL	R X J-	$y <$ MRL MRL $< y <$ AL $y >$ MRL and $y >$ AL

Notes: 1. The project-required holding time limit and the calculated holding time for the sample are denoted by HTL and t, respectively. 2. The concentration of the field sample, the action limit, and the method reporting limit are denoted by y, AL, and MRL, respectively.